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PRESS BULLETIN No. 35.

Sisal and the Utilization of Sisal Waste.

By E. V. Wilcox

AND

Wm. McGeorge

CULTIVATION AND ECONOMIC RELATIONS OF SISAL.

By E. V. Wilcox.

Bulletin No. 4, published by this station in 1903, covered rather completely the botanical aspects of sisal, the various processes connected with planting, cultivation, harvesting, milling and baling, together with notes on insects and diseases of this crop. The supply of the bulletin has long been exhausted and frequent requests have been received for further literature on sisal in Hawaii. The particular occasion for the chemical investigations reported in the present bulletin arose from the desirability of learning what economic use could be made of the large quantity of pulp which accumulates about the mill from decortication of the sisal leaves. In connection with this chemical work a fresh review was made of the conditions of the sisal industry in Hawaii at the present time.

Sisal was first brought to Hawaii from Florida in 1893. After a preliminary test at Ewa, Oahu, the plant was thought worthy of trying out on a large scale. The Hawaiian Fiber Company was organized for this purpose and has continued the production of sisal since that time. The plant was found also to thrive well on the other islands. Plantings of considerable size were made on Kauai, Hawaii and Maui. On account of the pressure of other industries, however, it was not found desirable to continue sisal production on the other islands, particularly since it was impossible to secure sufficiently large areas which were not adapted to other crops promising a rather larger profit. At present the Hawaiian Fiber Company is practically the only commercial organization engaged in the production of sisal in Hawaii. As just stated, local economic reasons weigh against the great extension of the industry on other islands.

The area at present under cultivation on Oahu is about 2,000 acres, located partly at Sisal and partly at Robinson. There is little possibility of further extension at Sisal, while at Robinson the opportunity for enlarging the area of sisal is fairly good. The profit per acre derived from the cultivation of sisal is rather small as compared with many other tropical crops, and for this reason there is greater need for the cultivation of the crop in large and continuous areas in order to secure reasonable profits. It is generally considered that areas of less than 1,000 acres are not likely to yield profitable remuneration when planted to sisal. Much larger areas, up to 10,000 acres or more, give better promise.

The present output of fiber from the mill located at Sisal is from one-half to one ton per day, depending on whether one or two machines are running. Recently a third machine has been started in operation in the Robinson section of the plantation and the total output will thus be increased accordingly. The baled fiber is an unusually beautiful product, being well cleaned and laid straight and at full length in the bale, so that no kinks are produced in the fiber. The quality of Hawaiian sisal is equal to the very best. In fact, the only sisal which brings an equal price is that which is produced in German East Africa. Recently, for reasons not perfectly understood, the price of sisal fiber was driven down to 4 cents per pound, but it has again recovered to the extent of 1 cent.

The annual yield, as judged by the results thus far obtained by the Hawaiian Fiber Company, is about 500 pounds of fiber per acre. The sisal plant matures under Hawaiian conditions in from three to four years, and sends up poles at the age of six to nine years. The true sisal seems on the whole to possess qualities making it superior to henequen for Hawaiian conditions. The life of the sisal plant is not so long as that of the henequen, but the quality of the fiber is better and the ease of handling is greater on account of the absence of spines along the edges of the leaves.

Among the more striking results obtained from the practical experience in the growth of sisal on Oahu is the fact of the great advantage of cultivation. If the sisal sucker at proper age and size for planting is merely placed in holes dug in the ground at the usual spacing distance, eight or nine feet, without previous plowing of the soil, the growth is slow as compared with that after plowing the soil. It seems not to be necessary to cultivate the soil after planting, if thorough plowing has been done before planting. At any rate, by merely plowing without subsequent cultivation one full year is cut off from the time required to reach maturity. This is plainly an important matter in the economy of sisal production. In addition to the saving of one year's time, a further advantage is obtained from plowing in that the plants make a more vigorous growth and produce leaves of greater size and length, thus increasing the total amount of fiber per acre. Mr. Wm. Weinrich, Jr., manager of the Hawaiian Fiber Company, has made a careful study of the economy of plowing versus not plowing, and the results indicate clearly the advantage of plowing. There is a still further advantage from plowing, in that corn and other crops may be cultivated between the rows of sisal for the first two years after planting sisal suckers.

Sisal is perhaps the best crop to be recommended for the large area of dry, highly manganiferous soil on the Wahiawa Plains. As has been clearly demonstrated, pineapples do not thrive on soil containing more than 2 per cent of manganese; in fact, even a lower percentage may cause a very unsatisfactory growth of pineapples. On some of this soil corn and other crops make a fairly good yield so long as the normal rainfall occurs and cutworm and leaf hoppers are not too abundant. Many almost

complete failures have occurred, however, in trying to raise intercrops in the sisal fields on account of the prevalence of insect pests or the lack of sufficient rain. There is no difficulty connected with the growth of sisal on this dry, highly manganiferous soil. Sisal in its behavior to manganese presents a striking contrast to pineapples. Apparently the reason for the injurious effect of manganese on pineapples is to be found in the fact that the manganese causes an undue absorption of lime and a reduction in the amount of magnesium and phosphoric acid. All pineapples have been shown to be very susceptible to an excess of lime. Manganese exercises the same effect upon sisal as upon pineapples, but the sisal is a lime-loving plant and is not disturbed by the presence of unlimited amounts of lime; in fact, at Sisal the plants are growing in coral limestone with only a mere trace of soil which has been washed down from higher levels.

One of the unexpected results obtained through the chemical examination of sisal is the fact that the normal acid of sisal is lactic. It was at first suspected that perhaps the presence of lactic acid might be a part of the basis of its reacting toward manganese so differently from pineapples. This supposition, however, was not borne out by further investigation, since manganese was found to be fully as soluble in the lactic acid of sisal as in the citric acid of pineapples. While lactic acid has been occasionally found in the seeds of certain plants, sisal is apparently the first plant to be shown to develop this acid as its normal vegetable acid.

The chemical investigations reported in this bulletin will indicate pretty clearly the prospects for the economic utilization of sisal pulp or waste. About 2,000 tons of this pulp has accumulated in the dump heap behind the mill at Sisal, and about 25 tons of pulp are being added per day. In estimating the moisture content of the pulp after standing, it appears safe to reckon it at about 50 per cent. In milling at Sisal it has been found necessary to use water in order to prevent a stickiness and improper cleaning of the fiber. At Robinson the water content of the leaves is somewhat higher and no water is required in milling. Even where water is used in milling, however, it rapidly runs out of the pulp in milling and does not add appreciably to the moisture content of the pulp on the dump heap. Assuming the

moisture content of the pulp on the dump to be 50 per cent, the actual fertilizer value of this pulp is about $6.00 per ton. As the chemical investigations indicate, the amount of mineral plant food in the sisal waste is rather high. In addition to its being a mineral fertilizer, there are the nitrogen and humus values to be considered. The feeding experiments that have thus far been made give only slight encouragement to the idea of using the pulp as a cattle feed. Cattle do not apparently take kindly to it, although its aroma and flavor, especially when dried are rather agreeable. It is evident from the chemical investigation of sisal that the value of the plant food in the pulp for fertililzer purposes is greater than the value for alcohol production of the fermentible substances.

The prospects for sisal production on the island of Oahu are at present very satisfactory. Practically all of the milling difficulties have been overcome. The known corrosive effect of lactic acid upon metals gave considerable difficulty at first, but the sprocket chains and other exposed parts of the machine if made out of gunmetal resist the action of the acid in a satisfactory way. There are at present no serious diseases or insect pests to contend with in growing sisal, at least on the island of Oahu. A peculiar disease of unknown nature, causing a discoloration of the fiber, was observed on Hawaii, but has not occurred on the plantations of Oahu.

CHEMISTRY OF SISAL.

By Wm. McGeorge.

In the process of decortication of the sisal leaf, since the leaves contain only 4-5 per cent of fiber, there necessarily accrues a large amount of waste. For several years the planters of Yucatan, German East Africa, Java, Hawaii and other countries where sisal is grown commercially, have been searching for some possible means of an industrial utilization of this enormous waste. At the solicitation of the Hawaiian Fiber Company, this station undertook a study of the chemical nature of the sisal plant, to ascertain, if possible, some commercial use for their waste. So far as the author has been able to ascertain from the literature at hand, there has been published little thorough chemical work on the sisal plant. F. H. D'Herelle[1] carried on thorough investi-

1 Journal d'Agriculture Tropicale, June, 1910.

gations in Yucatan at the solicitation of the Mexican Government. Others have published analyses of the waste to determine its fertilizing constituents, and the Experiment Station at Mauritius has made several unsuccessful attempts to ferment the waste for the production of alcohol.

The economic uses of the various species of Agave date back many years and the methods of utililzation vary between wide limits. The inhabitants of the Central American countries and Mexico took advantage of its saponin content by using it for washing purposes, the fiber for making ropes and stringed musical instruments, the juice as an insecticide and the leaves as fodder for cattle. Agave americana, as well as several other species, at the time it is ready to send up its flowering stalk experiences a remarkable upward flow of sap. This is sometimes collected by cutting out the central bud and leaves and inserting a receiving vessel. By fermentation of this juice the natives prepared an alcoholic drink called pulque.

The two varieties cultivated in Hawaii and the ones to which our studies were mostly confined are sisal and henequen. Our laboratory investigation was confined to the possible use of the residue as fertilizer, as stock food or for the manufacture of industrial alcohol. A short study was also made on the general chemical composition of the plant, and how this composition may vary when affected by different weather and soil conditions; also to determine the nature of the organic acid which causes its strong acidity, and the possible commercial importance of this acid.

FERTILIZER VALUE.

The waste from the mill of the Hawaiian Fiber Company has been accumulating since the beginning of the industry, and at present there is a large quantity of it piled up at the mill. On the first visit of the author to the mill samples were taken from this pile with the view of ascertaining its fertilizing value, as that is undoubtedly the only means of its disposal in this decayed condition. Samples were also taken of the juice coming from the mill and fresh waste pulp as it was being hauled from the mill to the dump. The following table gives the results of the analyses:

ANALYSES OF WASTE PRODUCTS.

Sample No.	Nitrogen	Potash	Phosphoric Acid
	Per Cent	*Per Cent*	*Per Cent*
1.	.45	5.21	2.79
2.	1.35	1.70	1.23
3.	.88	.69	1.20
4.	1.04	1.70	.95
5.	.49	6.38	.67
6.	.93	2.19	.46

No. 1 is a sample of ash taken from the pile (the waste had been partially burned several times). No. 2 represents waste which had been in the pile about six months; No. 3, about one year; No. 4, about two years. No. 5 is a sample of brown powder obtained by evaporating the juice to dryness, and No. 6 is a sample of fresh waste pulp. These analyses are all on air dry samples, and are a fair representation of the plant food stored up in the pile, and clearly indicate the advisability of its being returned to the soil and the possibility of using the fresh waste as fertilizer. Application of the fresh waste would of course mean an addition of nitrogen and humus forming material to the soil, but would offer difficulties in application and transportation. The burning, or at least partial burning, of the waste would mean a loss of a large percentage of nitrogen and organic matter, but this would possibly be offset by the increase in content of mineral constituents and ease of transportation. As shown in the analysis of the ash, which was only partially burned, some of the nitrogen remains in the ash. By returning the waste to the soil in any form, lime, magnesia, potash and phosphoric acid, minerals on which sisal feeds most heavily, will be returned to the soil. Otherwise the land will in time tend to become exhausted.

FEEDING VALUE.

The use of this waste as stock food presents a possible means of its disposal. From the analyses of the sisal leaves the protein content varies from 0.73 per cent to 1.16 per cent on the fresh

leaf.[1] The following table shows the minimum and maximum percentages found in our analyses for constituents of feeding value:

FOOD ANALYSIS.

	Minimum	Maximum
	Per Cent	*Per Cent*
Moisture	80.22	88.00
Protein	0.73	1.16
Nitrogen free extract	7.58	13.57
Fiber	2.27	3.32
Ash	1.42	1.73
Ash Ingredients	Per Cent in ash	Per cent in leaf
Potash	10.08—20.64	0.17—0.35
Lime	20.08—45.68	0.36—0.79
Phosphoric acid	1.54— 9.76	0.04—0.14

A qualitative test failed to show the presence of more than a mere trace of fat or oil in the leaf.

The present location of the mill, upon a barren area of coral land, where it seldom rains, would probably necessitate the transportation of the waste, should it be found to be of practical food value. This condition would necessitate its being brought to a dried form, which could be done without materially altering its palatableness and would decidedly increase its food value. The air dried pulp has a very agreeable aroma and physical appearance which it will maintain indefinitely. In the air dried form it will contain about 5 per cent moisture, which state would increase its food value as follows:

Moisture	5.00	per cent
Protein	5.78	" "
Nitrogen free ext	60.01	" "
Fiber	17.97	" "
Ash	11.24	" "

These figures represent the minimum values found in the

1 For comparison with other feeding stuffs of Hawaii, see Bul. No. 13 of this Station.

course of our analyses. In view of the above facts, the dry waste appears the more suitable form to feed and is worth a fair trial as such, especially in view of the scarcity of forage crops in Hawaii.

CHEMICAL INVESTIGATION.

Since very little chemical work has been done on sisal, it was considered of sufficient importance to make such an examination, especialy to investigate the carbohydrate content with regard to its utilization for fermentation purposes. The carbohydrates to be found in vegetable matter include various sugars, starch, gums, and celluloses. Heretofore, in a great many of the analyses of vegetable matter these carbohydrates have been classified under "nitrogen free extract," a term used quite indiscriminately and having a very indefinite meaning. During late years various articles have been published and numerous reasearches made upon the composition of this nitrogen free extract. W. E. Stone carried on a great deal of work in this line and published a method[1] for the separation and determination of the carbohydrates. This method, slightly modified, proved to give good results when applied to sisal, and was chosen for use in the analyses made in this investigation.

EXPERIMENTAL.

Before beginning the actual analysis a preliminary examination was made to ascertain the character of the ether, alcohol and water soluble matter. About 100 grams of finely chopped leaf was covered with ether in a 500 cc. Erlenmeyer flask and allowed to stand one week with frequent shaking. The extract was filtered off and the residue washed by decantation several times with ether. The extract had a green color due to dissolved chlorophyl, acid reaction and bitter taste. No fat or oil was detected, and on evaporating off the ether the residue was composed largely of a green waxy or resinous substance and contained no crystals. The per cent of solid matter extracted by the ether was very small and contained no sugars.

The residue from the ether extraction was treated in a like manner with 95 per cent alcohol, filtered and the residue washed by decantation. The result of this extraction was a greenish

1 Jour. Am. Chem. Soc., Vol. XIX., p. 183.

brown solution with slightly bitter taste and acid reaction. On evaporation to dryness there was deposited a greenish black residue of a gummy nature. This alcoholic solution when evaporated to dryness and the resulting residue treated with cold water was found to be almost completely soluble. The water dissolved the gums and sugars, both reducing and nonreducing, and the acid, as on redissolving the residue insoluble in water it gave a neutral reaction with litmus. The residue insoluble in water contained no sugars as it failed to reduce Fehling's solution either before or after inversion.

The residue from the ether and alcohol extraction was next treated in a like manner with water. The solution obtained had a yellow color, acid reaction and fairly agreeable taste. Upon standing several days this solution developed a moldy scum, indicating its ready susceptibility to bacterial growth. It contained reducing and nonreducing sugars and a relatively high per cent of gums. On evaporation to dryness the resulting residue was insoluble in alcohol, benzene, slightly soluble in ether, and formed an emulsion with chloroform. From the above examination it was considered to be unnecessary to make an extraction with ether in the analyses, but it seemed imperative to use the alcohol extraction followed by one with water and then treatment with diastase for the starch determination. It is evident that there is present in the leaf a small percentage of reducing and nonreducing sugars and a large percentage of gums which are insoluble in alcohol and would be acted upon by the acid inversion and subsequently determined with starch, thus giving too high a percentage of starch. This danger is eliminated by extraction with water before treating with diastase.

METHOD OF ANALYSIS.

1. Several fresh leaves were cut up into small pieces and from this 100 grams were weighed into a 1-litre flask fitted with a reflux condenser. Five hundred cubic centimeters of 95 per cent alcohol were added and the whole boiled for three hours. After thoroughly cooling, preferably letting stand over night, the alcohol was filtered off and the residue in the flask washed several times by decantation. This solution was evaporated on the water bath almost to dryness and then treated with water.

By this means the water dissolved the sugars and gums, leaving a residue of resin. The contents of the dish were then filtered through asbestos with the aid of a filter pump and the filtrate made to 200 cc. The resins remaining on the asbestos pad were dissolved in alcohol and the amount determined by evaporating an aliquot to dryness in a tared porcelain dish and weighing the residue. Next the reducing and nonreducing sugars were determined in aliquots of the 200 cc., by titration against Fehling's solution before and after inversion. Total solids was determined by evaporating an aliquot of this solution to dryness in a tared porcelain dish. The solids in solution other than sugars were considered to be gums and were thus determined by difference.

2. To the residue from the alcohol extraction 300 cc. of distilled water was added and allowed to stand at room temperature for 24 hours with frequent agitation. The residue was then filtered off and the solution was made to 500 cc. The reducing and nonreducing sugars were determined with Fehling's solution before and after inversion. Total solid matter was determined in an aliquot of the solution as in No. 1 and gums were obtained by differencce.

3. The residue from the aqueous solution was brought to an air dry condition and its weight accurately determined in order to establish its quantitative relation to the original weight of the leaf. Two grams of this residue were weighed into a 250 cc. beaker, 100 cc. of water added, and the whole boiled for one hour to convert the starch into a soluble form. This was then cooled to 65° C. and 25 cc. of a strong infusion of malt added. The malt was then allowed to act on the starch for several hours at 65° C. and subsequently allowed to remain in a warm place over night. This solution was filtered, the residue washed upon the filter paper and the solution evaporated to 100 cc. This was transferred to a 250 cc. flask, 10 cc. of concentrated hydrochloric acid added and the flask fitted with a reflux condenser and placed in a boiling water bath for one hour. It was then nearly neutralized, brought to volume and its sugar content determined against Fehling's solution. A blank determination was made upon the malt extract and results corrected accordingly.

4. The residue from the malt extraction was removed from the filter, 100 cc. of water and 2 cc. of concentrated hydrochloric

acid added and the whole boiled under a reflux condenser for one hour. The pentosans being in this manner converted into reducing sugars were determined by nearly neutralizing, bringing to definite volume and titrating against Fehling's solution.

5. The moisture, ash, crude fiber and nitrogen determinations were made upon the fresh leaf. Moisture and ash were determind on the same sample by heating 5 grams in the air bath at 100° C. for five hours. The loss in weight was considered as moisture. On this residue the ash was determined by ignition. Crude fiber was determined by drying 2 grams of the fresh leaf in an air bath and treating acording to the Official Method. Nitrogen was determined by the Kjeldahl Method.

The following table gives a comparison of the different samples analyzed, and attention is called to their variation in carbohydrate content:

CHEMICAL COMPOSITION OF LEAF.

	SAMPLE NUMBERS					
	1.	2.	3.	4.	5.	6.
	Per Cent	*Per Cent*	*Per Cent*	*Per Cent*	*Per Cent*	*Per Cent*
Reducing Sugars	12.49	18.68	10.45	20.03	7.48	18.50
Nonreducing Sugars	5.58	17.32	27.77	7.56	7.33	2.69
Gums	35.14	15.67	23.48	23.72	34.58	38.51
Starch	.66	2.57	.66	1.80	.64	1.25
Pentosans	2.32	4.92	3.96	4.73	5.95	4.58
Nitrogenous matter	7.25	4.75	4.89	6.00	3.63	5.19
Resin	2.82	.83	1.84	1.53	1.57	1.93
Fiber	20.86	23.13	15.80	23 71	26.49	18.32
Ash (total)	14.42	8.53	9.16	9.14	11.83	14.76
Acidity	.....	4.42	3.48	3.94	1.56	.....

In this table column 1 is the analysis of a sample of sisal grown at Robinson, Oahu, on a red clay soil; 2 is that of a sample grown on the station grounds, where it received much rain and in a tufa soil; 3 that of a sample grown at Sisal, Oahu, on coral land with very little rain; 4 that of a sample of hene-

quen grown on the Experiment Station Grounds; 5 that of henequen grown at Sisal, and 6 is the analysis of a sample of Agave americana grown on the Experiment Station Grounds. It is thus seen under what varying conditions sisal may be grown with apparent success.

It may be well at this point to insert a table showing the amount of sugars and gums in the fresh leaf.

	1	2	3	4	5
	Per Cent	*Per Cent*	*Per Cent*	*Per Cent*	*Per Cent*
Moisture	88.00	86.20	81.01	84.30	80.22
Sugar	2.16	4.97	7.25	4.33	2.93
Gums	2.97	1.28	3.36	2.59	5.84

The columns are numbered as in the preceding table.

DISCUSSION OF METHOD AND RESULTS.

1. The extraction with alcohol proved to be a reliable means of dissolving the larger part of the sugars occurring in sisal, which were found to be in large part sucrose, levulose and dextrose. It was found absolutely necessary to allow the alcohol to thoroughly cool before filtering off, for the reason that in so cooling a flocculant precipitate settled out. This was, no doubt, saponin, and if the solution was filtered hot it would precipitate in the filtrate as the solution cooled. Again, in the determination of the sugars by titration against Fehling's solution it was found to be advantageous and in most cases imperative that the gums be removed by precipitation with lead acetate and subsequent removal of the lead with sodium carbonate before titrating against Fehling's solution. Although this procedure consumed considerable time, if it was not followed out a yellow colloidal precipitate was formed when the sugar solution was added to the copper solution. This failed to separate out on prolonged boiling and in this manner obscured the decolorization of the Fehling's solution. On filtering, to test the filtrate for unreduced copper, this yellow colloid passed directly through the filter paper carrying with it reduced copper. By precipitation of the gums with lead acetate and subsequent removal of the lead with sodium

carbonate, this trouble was entirely eliminated. However, in cases where a titration could be made without treatment with lead acetate it was found on making a comparison of the results from titration of a solution treated as above and the same solution untreated, that the latter showed a slightly higher sugar content. In no case was the author able to get results with the polariscope which compared favorably with the results obtained by titration.

2. In every case it was found that the extraction with cold water dissolved sugars which were apparently insoluble in alcohol. Treatment with lead acetate before titration was found to be more imperative in this case than in that of the solution in alcohol, probably due to the fact that the former solution contained a larger amount of gums. It is an interesting fact that the sum of the water and the alcohol soluble matter is a fairly constant quantity except in the case of Nos. 3 and 6, as shown in the following table:

WATER AND ALCOHOL SOLUBLE CONTENT.

	SAMPLE NUMBERS					
	1.	2.	3.	4.	5.	6.
Reducing Sugars—	*Per Cent*	*Per Cent*	*Per Cent*	*Per Cent*	*Per Cent*	*Per Cent*
Soluble in Alcohol	10.83	14.88	8.66	16.62	6.42	16.32
Soluble in Water	1.66	3.80	1.79	3.41	1.06	2.18
Nonreducing Sugars—						
Soluble in Alcohol	4.83	12.79	21.50	5.81	5.00	2.28
Soluble in Water	.75	4.53	6.27	1.75	2.33	0.41
Gums—						
Soluble In Alcohol	15.38	4.80	8.96	7.55	12.75	18.05
Soluble in Water	19.76	10.87	14.52	16.17	21.83	20.66

By reference to the table it may be seen that in the cases where the sugar content is high the gums are low and vice versa. In view of this fact several leaves were allowed to remain in the laboratory after being cut for about one month. Analyses were

made on the fresh leaf and after standing one month, with the following results:

DECREASE OF SUGAR CONTENT.

	Fresh leaf	Same one month old
	Per Cent	*Per Cent*
Reducing Sugars	18.68	2.97
Non-reducing Sugars	17.32	9.17
Total Sugars	36.00	12.14
Gums	15.67	39.53

Analyses are calculated to moisture free basis, and show the large decrease in sugar content of both reducing and nonreducing sugars, more marked in that of the former, and also the marked increase in amount of gums corresponding to the decrease in sugars. On the leaves which were allowed to remain in the laboratory for some time there was a gummy exudation at the base end. Coupled with this is the fact that it is the general practice at the mill to decorticate the leaf as soon after cutting as possible. Otherwise the fiber is not so easily cleaned. F. H. D'Herelle found that reducing sugars in the sisal leaf increased upon standing, little by little, and reached a maximum on the sixth day.

3. The sisal leaf, as may be seen from the table, contains only a very small percentage of starch. By treatment of a cross section of the fresh leaf under the microscope with iodine, blue spots were few and widely scattered. In view of this fact the solution was boiled vigorously, and after neutralizing a strong infusion of malt was allowed to act for some time to bring all the starch into solution. The solution obtained in No. 2 by treatment with cold water contained no soluble starch.

4. The residue from the starch extraction still contained the actual cellulose or crude fiber and the more complex carbohydrates, such as pentosans, which upon treatment with acid are hydrolyzed into sugars capable of reducing Fehling's solution. From comparative tests the treatment with acid was found to give more concordant results than the method of distillation with strong hydrchloric acid and subsequent determination of furforol obtained in the distillate.

5. In the determination of the crude fiber it was considered necessary to make this upon the original sample according to the Official Method. It was found that the analysis of the residue from No. 4 gave too low results.

CRUDE FIBER.

Sample No.	I	II
	Per Cent	*Per Cent*
1	23.13	17.56
2	23.71	18.30
3	24.52	20.77

Column I shows per cent of fiber obtained from three analyses of the fresh leaf by the Official Method and column II the per cent obtained by analysis of residue No. 4. It was found impossible to obtain good checks on duplicate analyses made by the latter method.

CHARACTER OF THE ACID IN SISAL.

It is a well known fact among those engaged in the decortication of sisal leaves that the juice is very destructive to the metal in the machinery with which it comes in contact and also to the hands of the laborers who handle the fiber as it comes from the machine. In view of this fact, it was considered to be of sufficient importance to attempt to identify this acid and ascertain if it chanced to be of commercial value. So far as the author has been able to ascertain from literature at hand, the character of the acidity has never been determined definitely. M. D'Herelle[1] calls attention to the strong organic acidity of the leaf, which he says is due in part to oxalic acid. I was unable to get a test for free oxalic acid from a solution obtained by boiling the leaves vigorously in water, but on treatment in like manner with hydrochloric acid a faint test for oxalate was obtained. As previously mentioned the ether, alcohol and water extracts all gave an acid reaction, but from none of these could any crystaline substance be obtained by concentrating on the water bath. Fermentation goes on very rapidly when particles of the leaf are steeped with

1 Loc. Cit.

about twice their weight of water, and even in the leaf itself if evaporation of its moisture content is prevented. By boiling about 500 grams of the leaf in water, acidified with sulphuric acid, and subsequently evaporating this solution to a small volume, a fairly concentrated solution of the plant acid was obtained. This was placed in a separatory funnel and extracted several times with ether, the ether evaporated off and the residue taken up in water. The resulting solution has the following properties:

1. Decolorizes potassium permanganate solution in the cold in acid, alkaline and neutral solution.
2. Bitter taste and strongly acid to litmus.
3. No visible color reaction with ferric chloride.
4. Acid is nonvolatile at boiling point of water. At this point it may be of interest to add that on placing 500 grams of the leaf in a copper retort and subjecting to destructive distillation, the distillate remained neutral until the retort reached 130° C., temperature at which lactic acid is decomposed.
5. Acid miscible with alcohol, ether and water.
6. In the fresh leaf this acidity increases on standing in water to a maximum and then decreases.

In addition to these tests a measured volume of fresh juice was titrated with N/5 potassim hydroxide and showed an acidity of 0.45 per cent. This neutralized solution was allowed to stand and after two days' time upon titration it showed an increase in acidity of 0.75 per cent, or a total acidity of 1.10 per cent. This procedure was continued for eight days, at which time the solution reached a maximum acidity. These properties indicated the identity of the acid as being lactic acid, and in view of this fact the above tests were repeated on leaves obtained from plants grown on the Experiment Station Grounds, the juice of which was extracted as soon after cutting as possible. In this manner was eliminated the possibility of lactic acids being formed by lactic fermentation, which is known to take place in silage, etc.

A sample of the acid thus obtained, when subjected to Uffelman's[1] test for lactic acid, gave a decidedly positive reaction. In view of the further fact that this reagent has recently been found to also react with oxalic, citric, tartaric and succinic acids,[2]

1 Zeit, klin. Med. VII, p. 392.
2 Abs. in E. S. R., Vol. XXIII, p. 115.

these acids were all carefully tested for with negative results. Another test for lactic acid, recommended by Georges Deniges,[3] namely, treatment with concentrated sulphuric acid and an alcoholic solution of guaiacol, gave a positive reaction. In this method the sulphuric acid converts the lactic acid into acetaldehyde, the presence of which is detected by means of the guaiacol.

Following these tests a portion of the acid was subjected to an analysis by the Palm method.[4] In this analysis the per cent of Pb O obtained by incinerating the lead lactate, 3Pb O2 (C3 H6 O3), was found to be 78.36 and 78.07 per cent in two precipitations. The theoretical per cent is 78.50. Crystals were also obtained by treatment with zinc oxide which resembled zinc lactate crystals under the microscope, but no chemical analyses were made of these crystals to positively identify them as such. The above results were considered sufficient to warrant the conclusion that the acidity of the sisal leaf is due in most part to lactic acid.

Soon after the completion of this work the Fiber Company placed a new mill in operation at Robinson, Oahu, where the new plantation is located. They immediately noticed that the juice from the leaves did not attack the machinery so vigorously nor the hands of the laborers. Also that the decortication could be carried out without the addition of water, which is undoubtedly due to the fact that the leaves in this district contain 5 to 10 per cent more moisture than those from Sisal.

Leaves were sent to the laboratory from the two districts and the acid content was determined, with the result that those from Robinson showed the higher acid content. However, these leaves were younger and shorter in length, and would necessarily show a higher acid content than the older leaves, as has been proven in our investigation and which is shown in the following table:

TABLE COMPARING LENGTH OF LEAF WITH ACID CONTENT.

	1	2	3	4
Length of Leaf.....	29 inches	38 inches	48 inches	50 inches
Acidity	.53 per cent	.51 per cent	.36 per cent	.27 per cent

3. Abs. Jour. Chem. Soc., London, Vol. XCVI, pt. II, p. 627.
4 Jour. Amer. Chem. Soc., Vol. IX, p. 16; also Vol. XXXI, p. 1365.

Numbers 1, 2 and 3 are leaves from Robinson and 4 is from Sisal, and from the data at hand I do not believe there is any radical variation in acid content between leaves of the same length from the two districts. However, it has been noticed that the juice from leaves grown on the coral soil ferment much faster in the laboratory. After neutralizing the acid in the juice from 40 grams of the leaves, the whole was allowed to stand over night and titration repeated, with the result that the leaves from coral soil showed an increase of 0.31 per cent, while the leaves from Robinson had increased only 0.14 per cent. This may possibly be due to the higher lime content of the former, since lactic fermentation takes place more readily in presence of lime, and may offer some explanation as to why the machinery is attacked more vigorously at the Sisal mill.

The fact that the shorter leaves showed a higher acid content than the longer ones led the author to investigate the cause of this variation, since the only difference lies in the fact that the stump end of the leaf is much larger in proportion than in the smaller leaf and consequently contains more juice. A leaf, four feet long, was taken and cut into four lengths, each one foot long, and the acid content of each length determined with the following results:

TABLE SHOWING VARIATION IN ACID.

	1	2	3	4
Per Cent Acid	0.64	0.61	0.34	0.15

The samples are numbered beginning with the tip, which is No. 1, and ending with No. 4, which is the one-foot length nearest the stump of the plant. These results offer an explanation as to why the younger leaves contain the higher acid content.

INDUSTRIAL ALCOHOL FROM SISAL WASTE.

The analyses for carbohydrate content were made primarily with the idea in view of ascertaining its profitableness for making industrial alcohol. M. D'Herelle[5] considers that the *Agave rigida*

5 Loc. Cit.

Var. Sisalana of Yucatan can be used profitably for the production of alcohol and that other varieties of agave may also be used with just as good results, in consideration of the fact that the raw material is free from cost. He considers the industrial maximum as being 16 litres per 1,000 leaves and the average as being 13 litres per 1,000 leaves. With the daily working of 175,000 leaves in Yucatan they would obtain about 575 gallons of 95 per cent alcohol per day. With this large quantity of juice to work on and having overcome the difficulties to be encountered in fermenting sisal waste, the industry may be profitable enough to pay for the chemist or bacteriologist necessary for the supervision of the plant, thus forming a remunerative industry. In his article he cautions others as to the difficulties of the fermentation of sisal waste, and states that after trying a number of species of yeasts obtained from the Pasteur Institute, he finally provoked a successful fermentation with a special kind of yeast, the identity of which he does not divulge. Other experimenters[1] have not been so successful.

In view of the comparatively small quantity of waste accruing at present at the Hawaii plantations and the very low sugar content of sisal leaves, it is not deemed a profitable undertaking to utilize the sisal waste of Hawaii for making alcohol, taking into consideration the difficulties to be overcome in the fermentation. Several fermentations were made by the author in this laboratory, and while an alcoholic fermentation was obtained by using ordinary compressed yeast, the yield was very poor and indicated the difficulties to be overcome. In one instance an alcoholic fermentation was obtained by simply adding an excess of calcium carbonate and then allowing to ferment by mere exposure to the air, the calcium carbonate combining with the lactic acid as it is formed, thus checking the lactic fermentation.

In our analyses the sugar content of the fresh leaf varied from 2 to 7 per cent, making an average of 4.5 per cent,[2] which theoreticaly would yield about 26 litres (or 6.5 gallons) absolute alcohol per ton of leaves. It is hardly conceivable that with such

1 Bul. No. 25, Sta. Agron. Colony of Mauritius.

2 These samples being taken from different parts of the island and grown under varying conditions, give a fair average of the sugar content of Hawaiian sisal.

a sugar content this could be made a remunerative industry with the waste at hand, and even with the large quantities of waste in Yucatan it would require a rather efficient system. The actual recovery of the juice, which is only about 50 per cent of that in the waste, and the services of a man trained in fermentation industries and familiar with the properties of sisal waste are two important factors to be considered before undertaking the manufacture of industrial alcohol from the sisal waste.

For comparison it may be of interest to show the carbohydrate content of other fermentable substances along with sisal leaves:

CARBOHYDRATE CONTENT OF SOME FERMENTABLE SUBSTANCES.[1]

Corn	70	per cent
Potatoes	20	" "
Cassava	30 - 40	" "
Sweet Potatoes	20 - 25	" "
Bananas	20	" "
Cactus	7 - 20	" "
Sisal (our analysis)	2 - 7	" "

Herbert and Heim,[2] after working on sisal waste, concluded that while the fermentation of sisal waste for alcohol deserves consideration, the obvious and immediate application of this waste is to return it to the land in the form of manure.

CHARACTER OF THE MINERAL CONTENT.

From a previous table, showing the analyses of the leaves, it may be seen that there is considerable variation in ash content, namely from 8.53 per cent to 14.76 per cent on the moisture free material. This ash content is rather high in comparison to the most plants, and indicates the extent to which the sisal plant draws upon the soil for its mineral plant food. At this point will be given a table showing analyses of the ash obtained by burning leaves taken from various sources and growing under varying soil and climatical conditions.

1 Bul. 130, Bur. of Chem. U. S. Dept. of Chem.
2 Compt. Rend. 1909, 148, pp. 513-514.

CHEMICAL COMPOSITION OF ASH.

	1	2	3	4	5	6
	Per Cent	*Per Cent*	*Per Cent*	*Per Cent*	*Per Cent*	*Per Cent*
Silica ($Si O_2$)	.46	.84	.42	1.06	.74	.30
Ferric oxide ($Fe_2 O_3$)	1.18	.44	1.68	.51	.36	1.32
Aluminum oxide ($Al_2 O_3$)	4.86	3.80	4.04	4.40	5.66	4.74
Manganese oxide ($Mn_3 O_4$)	0.08	0.05	0.14	0.17	0.00	0.28
Lime (Ca O)	20.88	21.58	45.68	22.84	45.44	21.56
Magnesia (Mg O)	10.44	22.00	8.48	26.60	5.24	24.56
Potash ($K_2 O$)	20.64	12.00	10.08	14.68	10.64	17.80
Soda ($Na_2 O$)	5.68	10.12	3.68	1.96	5.24	6.44
Sulphur Trioxide ($S O_3$)	2.88	2.64	3.12	2.56	2.56	1.60
Phosphoric Acid ($P_2 O_5$)	1.54	8.57	3.50	9.76	4.40	6.24
Chlorine (Cl)	.....	.35	.....	.22	.....	.....
Carbon dioxide ($C O_2$)	31.36	17.61	19.18	15.24	19.72	15.16

The columns are numbered in agreement with the leaves, the analyses of which are reported in the table on page 12; i. e., column 1 is the analysis of the ash obtained from the ignition of the leaf whose analysis is shown in column 1, page 12. From these results it is clearly evident that the plant feeds most heavily upon phosphates, lime, magnesia, potash and soda, and since nearly all this mineral matter remains in the waste the analyses strongly indicate the advisability of returning the waste to the soil as fertilizer. Without this precaution the necessity of the application of artificial fertilizer to obtain best results is apparent.

From these results it may also be seen that there is a striking variation in the amounts of the minerals on which it feeds most heavily. Phosphoric acid in every instance was highest in those plants grown on the Station Grounds, varying from 6.24 to 9.76 per cent in the ash. The next highest were the plants grown at Sisal, Oahu, while the lowest, 1.54 per cent, was grown at Robinson, Oahu. The soil at the latter location is of the heavy, red clay type, the phosphoric acid content of which is known to be held in a difficultly available form as ferric or titanic phosphates. This fact indicates that better results could be obtained by applying small quantities of phosphoric acid in

a more available form. The fact that the plants grown on the coral soil contain more phosphoric acid is probably due to its being in a more available form than that in the heavy clay soil, namely, in combination with the lime. The character of the soil on which the plants are growing at the Experiment Station is a fairly loose soil, formed partly by the disintegration of the black volcanic ashes which make up the subsoil.

Similarly the lime content varies greatly according to environment, being of course highest in those plants grown on coral but showing little variation in the plants grown on the other soils. There is a rather striking relationship between the lime and magnesia contents of all the plants, namely a high percentage of lime correlating with a low per cent of magnesia and vice versa, and is apparently not accompanied by any noticeable injury to the plants. Those grown on the coral soil contain more than twice as much lime as the other plants, and considerably less magnesia in spite of the fact that Hawaiian corals contain from 0.11 per cent to 9.95 per cent magnesia.[1] It may be of interest in this connection to call attention to the magnesia content of the ash of plants grown at the station grounds and those at Robinson, Oahu, where the lime and magnesia content of the soils is as follows:

	Station	Robinson
	Per Cent	*Per Cent*
Lime	0.28	0.62
Magnesia	6.25	0.36

These results tend to indicate that the sisal plant when grown in an abundance of lime, will assimilate it in largest quantities but in normal soils will thrive just as well on smaller amounts of lime, replacing same with magnesia and in small part with potash as may be seen by reference to the table of ash analyses. The potash seems to be governed by the lime content but not nearly so strikingly as the magnesia. Manganese apparently has no injurious effect upon the sisal plant. The plantation at Robinson is situated in the district in which the highly magniferous

1 U. S. Geological Survey, Bul. 419, p. 210.

soils occur and the sisal plant is thriving and shows no deleterious effects from the manganese.

CONCLUSIONS.

1. Obviously the best utilization of the waste is to return it to the soil, for unless such is done the soil will in time tend to become exhausted. By using this waste as fertilizer all the mineral matter will be returned to the soil except that in the fiber, which will compensate for the most of the plant food removed from the soil by the plant.

2. The use of sisal waste as a stock feed has been fairly successful in other countries, when fed in the fresh form. I do not think it has ever been fed as a dry feed, but believe such a use to be fully deserving as a trial, not alone but mixed with other rations. When properly dried it contains a fairly high percentage of sugars from which it derives its nutritive value and should give good results along with other feeds.

3. The utilization of this waste for making alcohol is claimed by M. F. D'Herelle to be a remunerative industry in Yucatan. At present outlook I do not believe this means of utilization could produce promising results in Hawaii.

4. As for the lactic acid content, I do not consider that it could possibly be of any commercial value.

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